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TRANSLATION of Japanese Patent application No. Sho 63-30417 (A)

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10 Specification**1. Title: Anti-allergic agent****2. Claims**

15 (1) Anti-allergic agent having as the active principle a compound represented by the general formula

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25 wherein R_1 is a hydroxy group or O-D- β -glucose and R_2 is a methoxy group, or R_1 and R_2 together constitute a methylenedioxy group, R_3 and R_5 are hydroxy or methoxy groups, R_4 is a hydrogen atom or a methoxy group, R_6 is a hydrogen atom; a hydroxy or methoxy group.

30 (2) Anti-allergic agent in accordance with claim 1, wherein in the above general formula R_1 and R_2 are hydroxy groups, R_2 is a methoxy group, R_4 and R_6 are hydrogen atoms, and R_5 is a hydroxy group.

35 (3) Anti-allergic agent in accordance with claim 1, wherein in the above general formula R_1 and R_2 together constitute a methylenedioxy group, and R_3 , R_4 , R_5 and R_6 are methoxy groups.

40 (4) Anti-allergic agent in accordance with claim 1, wherein in the above general formula R_1 , R_3 and R_6 are hydroxy groups and R_2 , R_4 and R_5 are methoxy groups.

(5) Anti-allergic agents in accordance with claim 1, wherein in the above general formula R_1 is O-D- β -glucose, R_2 , R_4 and R_5 are methoxy groups, R_3 and R_6 are hydroxy groups.

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(6) Anti-allergic agent in accordance with claim 1, wherein in the above general formula R_1 is O-D- β -glucose, R_2 is a methoxy group, R_3 is a hydroxy group, R_4 and R_6 are hydrogen atoms, and R_5 is a hydroxy group.

5 3. Detailed Description

"Yakan" denotes the rhizome of the blackberry lily (*Belamcanda chinensis*), a plant of the Iridaceae family, which is known to have anti-microbial and anti-inflammatory effects. In their efforts to obtain medicaments against acute allergies, the present inventors among others examined the substances contained in this rhizome. In the process they have found an anti-allergic effect of the compound(s) having the general formula (hereinafter referred to as "compound of the general formula")

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20 (wherein R_1 is a hydroxy group or O-D- β -glucose, R_2 a methoxy group, or R_1 and R_2 together constitute a methylenedioxy group, R_3 and R_5 are hydroxy or methoxy groups. R_4 is a hydrogen atom or a methoxy group, R_6 is a hydrogen atom, a hydroxy or methoxy group) on which the present invention is based.

25 The compounds of the general formula may, for example, be obtained in the following manner. An extract of "Yakan", which is the dried rhizome of the blackberry lily *Belameanda chinensis* *Leman*, a plant of the Iridaceae family, is obtained by using a solvent selected from among water, methanol, ethanol, acetone either by itself or in mixtures with further solvents, heating from 0°C to a temperature below the boiling point of the used solvent, or by carrying out ultrasound extraction from 0°C to the ambient temperature. The extract may be left as it is, concentrated, dried, diluted by adding water, or a partitioned extract may be obtained by partitioning extraction using ethyl ether, benzene, chloroform, methylene chloride or the like.

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35 Moreover a partitioned extract is obtained by performing partitioning extraction with the aid of ethyl acetate, n-butanol, methylethyl ketone or the like.

The partitioned extract obtained in the above described manner may be left as it is, concentrated or dried, and the eluate is fractionated by subjecting it to column chromatography using silica gel, alumina, reverse phase silica gel etc. as a carrier. As the eluent it is possible to use an eluent selected from water or methanol, ethanol, acetone, acetonitrile, ethyl acetate, chloroform, methylene chloride, benzene, ethyl ether, n-hexane and the like, by itself or mixtures of other solvents. The fraction thus obtained may be left as it is, concentrated, or dried and is placed on a thin layer of silica gel including a fluorescent agent (e.g., RP-18 F254S by Merck or the like), and subjected to thin-layer chromatography by using a solvent selected from water or methanol, acetonitrile,

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tetrahydrofuran, acetone, ethyl acetate, chloroform, benzene, methylene chloride, ethylether and n-hexane by itself or mixtures thereof as a developer. After developing, the fraction containing the compound of the general formula separated by means of ultraviolet (254 nm) radiation is subjected to recrystallization by using a solvent selected from water, methanol, ethanol, tetrahydrofuran, ethyl acetate, ethylether, chloroform, acetone, methylene chloride, benzene and n-hexane or mixtures thereof, whereby the compound of the general formula may be obtained.

In the following, specific examples for the process of manufacturing compounds corresponding to the general formula are represented.

Tectorigenin

The compound in which R_1 , R_3 and R_5 are hydroxy groups, R_4 and R_6 are hydrogen atoms and R_2 is a methoxy group, is called Tectorigenin. It may be obtained in the following manner.

Example 1

2.5 kg of "Yakan" (rhizome of the blackberry lily *Belameanda chinensis* Leman) were pulverized and extracted for 2 h with 7 l of a 1:1 ethanol/water mixture while heated at reflux, the extract was filtered under heating, and the solvent was removed from the filter fluid under reduced pressure; four liters of water were added, and freeze-drying performed, whereby 428.61 g of extract (yield factor: 16.87%) was obtained.

This freeze-dried extract was diluted and dissolved with 1.5 l of water, partitioning extraction was performed 5 times with 2 l each of ethyl ether, the partitioned extracts were combined and the solvent removed under reduced pressure, whereby 41.20 g of ethylether extract (yield factor: 1.6%) were obtained. Moreover the partition aqueous solution was partition extracted 5 times with 5 l each of n-butanol, the partitioned extracts were combined, the solvent removed under reduced pressure, 1 l of water added and freeze-drying performed, whereby 122.69 g of freeze-dried extract (yield factor: 4.83%) was obtained.

Finally the 41.20 g of ethylether extract was subjected to column chromatography by using 400 g of silica gel (*Kieselgel* 60.70-230 mesh by Merck), and elution was performed by gradually increasing the proportion of ethyl acetate in the solvent mixture of benzene and ethyl acetate. From the fraction eluted with 4 l of solvent mixture of benzene and ethyl acetate (9:1) the solvent was removed under reduced pressure, and recrystallization was performed with a solvent mixture of n-hexane and tetrahydrofuran (1:1), whereby 2.22 g of a slightly yellow, needle-shaped substance (yield factor: 0.09%) was obtained.

This slightly yellow, needle-shaped substance conformed with the physical and chemical properties of Tectorigenin as described in literature [*Asa Kawanase, Naokazu Ohta, Kazuyoshi Yagishita; Agr. Biol. Chem.*, 37 (1) 145 (1973)].